

IN THE CLAIMS:

1. (currently amended) A non-pressurized method for the continuous production of alkyl esters of higher fatty acids from fatty acid triglyceride starting mixtures containing a free fatty acid content of 50% to 100% free fatty acids, including an integrated combination of acid esterification and basic transesterification, said method comprising:

- a) at least ~~one~~ two esterification reactions of the free fatty acids of the fatty acid triglyceride starting mixture in separate fluidly connected esterification devices ~~connected with each other~~, such that free fatty acids in the starting mixture are esterified with a C₁-to C₄-mono alcohol in the presence of an acid catalyst and glycerine as a dragging agent, at 60° C. to 65° C., such that to produce an esterification mixture is produced,
- b) partial purification of the first esterification mixture via partial separation of the dragging agent, acid catalyst and unconverted C₁-to C₄-mono alcohol,
- c) transesterification of the fatty acid triglycerides, carried out at least twice, in separate transesterification devices connected with each other, with a C₁-to C₄-mono alcohol in the presence of a base catalyst at 60° C. to 65° C. to produce a transesterification mixture and
- d) purification of the transesterification mixture via separation of the basic catalyst, unconverted C₁-to C₄-monoalcohol and the glycerine produced during transesterification, by a treatment with water in at least one separator with subsequent drying, wherein the C₁-to C₄-mono alcohol used for esterification, the ~~conveying~~ dragging agent glycerin used for esterification and the water used for purification of the transesterification mixture are at least partially recovered from the esterification and transesterification mixtures and further wherein after purification the acid and base catalysts from the esterification and transesterification mixtures are converted, resulting in the production of a salt suitable for use as a fertilizer.

2. (currently amended) The method according to claim 1, wherein the fatty acid triglyceride initial mixtures containing free fatty acids are selected from the group

consisting of used and unused, unpurified and purified vegetable, animal and industrial oils and fats and mixtures thereof ~~with a free fatty acid content of 0% to 100%.~~

3. (original) The method according to claim 1 wherein the unpurified oils and fats are selected from the group consisting of soapstock, brown grease, yellow grease, industrial tallow, industrial lard, oil used for deep-frying, animal fat waste products, edible tallow, unpurified crude vegetable oils, unpurified animal fats and mixtures thereof.

4. (original) The method according to claim 3, wherein the unpurified crude vegetable oils are selected from the group consisting of rapeseed oil, soybean oil, sunflowerseed oil, palm oil, maize germ oil, cotton seed oil, palm kernel oil and coconut oil.

5-8. (canceled)

9. (original) The method according to claim 1, wherein the C₁-to C₄-mono alcohol used for esterification is methanol or ethanol.

10. (original) The method according to claim 1, wherein the acid catalyst used for esterification is sulphuric acid or p-toluol sulfonic acid.

11. (canceled)

12. (previously presented) The method according to claim 1, wherein the free fatty acids are esterified 2 to 8 times in separate columns consecutive to and connected with each other, depending on the fatty acid content of the starting mixtures, and wherein the esterification mixture obtained from one column is conducted from that column into the consecutive column and whereby, after the addition of a mixture containing a dragging agent, a mono alcohol and an acid catalyst, the esterification mixture is esterified again.

13. (canceled)

14. (currently amended) The method according to claim 12 wherein a part of the esterification mixture from one column is conducted to the consecutive column and whereby a part of the resulting esterification mixture is re-conducted into the a preceding column.

15. (previously presented) The method according to claim 1, wherein the free fatty acids are esterified 2 to 8 times in separate columns consecutive to and connected with each other, depending on the fatty acid content of the starting mixtures, and wherein the esterification mixture is partially purified prior to being conveyed into the consecutive column.

16. (currently amended) The method according to claim 15, wherein a mixture containing dragging agent, acid catalyst, unconverted mono alcohol and water produced during esterification is separated as the a heavy phase via phase separation from the esterification mixtures and is conveyed to devices for the purification and separation of the components of the ~~mixture~~-heavy phase.

17. (original) The method according to claim 16, wherein the heavy phase is conducted into a drying device for the separation of mono alcohol and water.

18-19. (canceled)

20. (previously presented) The method according to claim 17 wherein at least one of the mono alcohol and the mono alcohol-water mixture is conducted from the drying device into a rectification device for further purification, and wherein the mono alcohol purified in the rectification device, having a water content of approximately 1% to 2% suitable for use in columns due to a higher fatty acid content, is re-conducted from the rectification device into the esterification device.

21. (original) The method according to claim 17 wherein a partial flow of the mixture obtained in the drying device after separation of mono alcohol and water and containing dragging agent and acid catalyst, is re-conducted from the drying device into the esterification device, and wherein a partial flow of the above-mentioned mixture is conducted into an acidification device.

22. (previously presented) The method according to claim 1, wherein the esterification device is a column with or without ceramic or metallic packings or packings made of wire fabric, and wherein the esterification mixture obtained after esterification in the last esterification column is conducted into an extraction column and is subjected to extraction in that column utilizing pure mono alcohol or a mono alcohol-dragging agent mixture for the removal of non-esterified free fatty acid.

23. (currently amended) The method according to claim 1, wherein the esterification device is a column with or without ceramic or metallic packings or packings made of wire fabric, and wherein an esterification mixture is obtained on completion of the last esterification step in the last esterification column or on completion of the an extraction step in the an extraction column and whereby almost all free fatty acids have been esterified and whereby the above-mentioned mixture has an acid number of approximately 1 to 0.5 and a maximum water content of 0.5%.

24. (currently amended) The method according to claim 23, wherein the esterification mixture is conducted into a transesterification device connected to the last esterification column or to the an extraction column for the basic transesterification of the fatty acid glycerides.

25-28. (canceled)

29. (original) The method according to claim 24 wherein the C₁-to C₄-mono alcohol used for transesterification is methanol or ethanol.

30. (original) The method according to claim 24 the base catalyst used for transesterification is selected from the group consisting of potassium hydroxide, sodium hydroxide and sodium methyllate.

31. (canceled)

32. (previously presented) The method according to claim 24, wherein the transesterification mixture obtained in a column is subjected to preliminary purification. and wherein a mixture of mono alcohol, base catalyst and glycerine is separated as the heavy phase via phase separation from the transesterification mixture and is conducted for further purification and separation of the components to an acidification device and subsequently to a separator arranged consecutively to the acidification device.

33. (original) The method according to claim 21 wherein the heavy phase separated from the transesterification mixture is mixed in the acidification device with the partial flow of the dragging agent and acid catalyst mixture that was separated following esterification and conducted into the acidification device, wherein the partial flow is proportioned in such a way that the base catalyst of the heavy phase is neutralised and the heavy phase is sufficiently acidified.

34. (canceled)

35. (previously presented) The method according to claim 24 wherein the transesterification mixture obtained in a column is subjected to preliminary purification, and wherein the pre-purified transesterification mixture is conducted into a separator for further purification, and wherein a water-containing mixture comprising mono alcohol, soap, base catalyst and glycerine is separated in the separator from the transesterification mixture with water.

36. (Original) The method according to claim 35, wherein pH-conditioned water or buffered water is used.

37-39. (canceled)

40. (currently amended) The method according to claim 24 wherein the transesterification mixture obtained in a column is subjected to preliminary purification, to produce a pre-purified transesterification mixture and wherein the pre-purified transesterification mixture is conducted into a separator for further purification, and wherein the purified transesterification mixture of the last transesterification column is conducted out of the separator into at least one additional separator for further purification, and wherein the transesterification mixture from the at least one additional separator is conducted into a drying device where it is dried and purified, and wherein following purification in the drying device a product mixture corresponding to biodiesel and comprising mainly C₁-to C₄-alkyl esters of the free fatty acids and C₁-to C₄-alkyl esters of the higher fatty acids is obtained from the fatty acid triglycerides.

41. (canceled)

42. (original) The method according to claim 32 wherein the fatty acids formed during transesterification are partially separated from the remaining components of the water-containing mixture in the separator that is consecutive to the acidification device and are re-conducted into the esterification device.

43-48. (canceled)

49. (currently amended) The method according to claim 42 wherein the remaining components of the water-containing mixture are conducted from the separator into the a rectification device, and wherein the mono alcohol is separated in the rectification device from the remaining components of the water-containing mixture and is re-conducted to the esterification device in a purified state, wherein the water content of the purified mono alcohol is approximately 1% to 2%, and wherein the remaining components of the water-containing mixture are conducted from the rectification device into an evaporation device, and wherein the water is separated in

the evaporation device and is re-conducted into the separators, and wherein the mixture comprising glycerine, acid catalyst and base catalyst is conducted into a distillation device for further purification, and wherein acid and base catalyst are conducted from the distillation device into a thin-layer-evaporation device where the acid and base catalysts are converted, resulting in the formation of a salt suitable as a fertilizer.

50- 89. (canceled)

90. (new) The method of claim 1, further comprising:

subjecting the fatty acid triglyceride starting mixtures containing free fatty acids to at least one esterification step to yield a partially esterified mixture;

drying the partially esterified mixture to obtain a dried partially esterified mixture;
and,

subjecting the dried partially esterified mixture to at least two additional steps of esterification.

91. (new) The method of claim 1, further comprising:

extracting an esterification mixture with a pure mono alcohol in an extraction column.

92. (new) The method of claim 91, wherein the extracting the esterification mixture with the pure mono alcohol in the extraction column comprises removal of non-esterified free fatty acids to obtain an extracted esterification mixture.

93. (new) The method of claim 92, wherein the non-esterified free fatty acids are conveyed to an esterification column for esterification.

94. (new) The method of claim 92, further comprising:

conveying the extracted esterification mixture from the extraction column to a transesterification device arranged consecutively to and fluidly connected with the extraction column; and,

carrying out alkali-catalyzed transesterification of fatty acid glycerides in the extracted esterification mixture.

95. (new) The method of claim 1, wherein glycerin produced during transesterification is used as the dragging agent.

96. (new) The method of claim 35, wherein the water used in the separator to separate a water-containing mixture comprising mono alcohol, soap, base catalyst and glycerin from the transesterification mixture comprises water generated in the esterification mixture.

97. (new) An integrated combination apparatus for the production of biodiesel from fatty acid triglyceride starting mixtures containing free fatty acids comprising:

an esterification unit comprising at least two esterification columns for esterification of the free fatty acids with a mono alcohol;

a transesterification unit fluidly connected to the esterification unit and having at least two transesterification columns for transesterification of fatty acid triglycerides;

a first purification unit for the purification of biodiesel fluidly connected to the transesterification unit; and,

a second purification unit comprising at least one evaporation apparatus for the recovery of water connected by at least one feed line and at least one discharge line to the esterification unit, wherein the second purification unit is fluidly connected to the transesterification unit for the purification and separation of compositions used in at

least one of the esterification unit or the transesterification unit and for the purification and removal of the water used in the first purification unit;

wherein the integrated combination apparatus is configured such that the compositions used in the esterification unit and the transesterification unit are simultaneously purified and separated in the second purification unit and compositions used for the esterification are conducted to the esterification unit.

98. (new) The method of claim 12, wherein different amounts of acid catalyst are introduced into the separate esterification columns.